Standard Operating Procedure For Nutrient Sample Processing

LG212

Revision 04, January 2007

TABLE OF CONTENTS

Section Number	Subject	Page
1.0	**SCOPE AND APPLICATION**	**1**
2.0	**SUMMARY OF METHOD**	**1**
3.0	**SAMPLE HANDLING AND PRESERVATION**	**1**
4.0	**APPARATUS**	**1**
5.0	**REAGENTS**	**2**
6.0	**EQUIPMENT CONFIGURATION**	**2**
7.0	**PROCEDURE**	**2**
8.0	**SAFETY AND WASTE HANDLING**	**3**

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Standard Operating Procedure for Nutrient Sample Processing

1.0 SCOPE AND APPLICATION

1.1 This method is a description of the procedure used for nutrient sample processing of water samples collected on the EPA monitoring vessel *R/V Lake Guardian* and includes the filtration and preservation of the samples for subsequent analyses for chloride, dissolved reactive silica, total phosphorous, nitrate plus nitrite, total dissolved phosphorous, calcium, magnesium, sodium, and potassium (tentative).

2.0 SUMMARY OF METHOD

2.1 Water samples are collected at multiple depths using a Rosette-Niskin bottle sampling system and transferred to one-half gallon plastic (HDPE) containers. Within one to two hours, a portion of these samples is filtered and the filtrate is used to fill two 125-mL polyethylene bottles.

2.2 One of the two bottles and a third 125-mL polyethylene bottle of raw sample water are preserved with acid equivalent to one mL/L of concentrated sulfuric acid.

2.3 One 250-mL polyethylene bottle of raw water, preserved with 1:1 nitric acid, may also be collected for cation analysis.

3.0 SAMPLE HANDLING AND PRESERVATION

3.1 Samples are collected in new one-half gallon plastic (HDPE) containers.

3.2 These procedures are performed within two hours of collection.

3.3 Clean Nitrile gloves are worn while performing this procedure to prevent contamination from oils from the hands of the operators.

3.4 The labels for nutrient samples are color coded: the TP sample labels are yellow, the TDP/NO_3 sample labels are orange, and the Cl/Si sample labels are white.

3.5 Lab and field duplicates are separate filtrations, the lab duplicate prepared from the routine field sample, the field duplicate from the duplicate field sample. See Appendix B, *Quality Assurance Project Plan for the Great Lakes Water Quality Surveys*.

4.0 APPARATUS

4.1 Ship's vacuum pump, tank, and automatic cut-off system.

4.2 Vacuum regulator set to 5 inHg (never to exceed 7 inHg) with inline safety flask to prevent liquid from entering the regulator.

4.3 Four 500-mL plastic separatory funnels with two-hole rubber stoppers.

4.4 Four 300-mL polysulfone 47-mm magnetic filter funnels.

4.5 Four three-port, two-way valves, attached to top of separatory funnel allowing the funnel to be connected to either

5 psi vacuum or open to atmosphere.

4.6 Two filter forceps, one labeled "new" to replace filter holder with a new membrane filter and one labeled "old" to remove used filters for discarding.

4.7 Bottle-top reagent dispenser set to deliver 0.4 mL.

4.8 Supply of 47-mm diameter Sartorius 0.45-µm membrane filters.

5.0 REAGENTS

5.1 Sulfuric acid solution. With continuous mixing, add 150 mL of concentrated sulfuric acid to 385 mL of reagent water.

CAUTION: Concentrated sulfuric acid will produce immediate charring of skin and natural fibers like cotton, wool and paper. Very high temperatures are generated when sulfuric acid is mixed with water. Never add water to containers of sulfuric acid. This operation should be performed in a fume hood.

5.2 Nitric acid solution. With continuous mixing add 150 mL of concentrated nitric acid to 150 mL of reagent water.

6.0 EQUIPMENT CONFIGURATION

6.1 The vacuum regulator is connected to the ship's vacuum system via metal vacuum tubing. The other connections are made with flexible plastic laboratory tubing. The regulator is connected through the inline safety flask to four two-way valves which are each connected to one hole of a two-hole stopper in the top of the separatory funnel, such that the separatory funnel connection can be switched between vacuum and atmosphere. The other hole in the two-hole stopper holds the filter funnel. The combination of filter funnel and separatory funnel are supported in a rack such that the separatory funnel can be conveniently drained into a 125-mL bottle.

6.2 The bottle-top reagent dispenser is set to deliver 0.4 mL of the sulfuric acid solution (5.1). It must be placed in a secondary containment vessel, within a fume hood, and in a secure area to preclude being overturned by the motion of the ship or by accidental contact.

7.0 PROCEDURE

7.1 Clean Nitrile gloves must be worn while working with the filtering equipment.

7.2 Prior to each monitoring survey, the filter funnels and separatory funnels are rinsed thoroughly with tap water and a laboratory brush, followed by rinsing with reagent water. Between stations, the filter funnels are covered with inert plastic covers. At the conclusion of each filtering operation, filter forceps are used to replace the dirty filter with a fresh filter.

Note: Remove dirty filter but do not replace with a fresh filter if the ship is in transit to another lake or if there will be a significant delay in nutrient sample processing. The Chief Scientist should be consulted if the analyst is unsure of whether or not a fresh filter should be placed on the filtering equipment.

7.3 For each nutrient sample, the filter cover is removed from the filter funnel, the separatory funnel stopcock is closed, and the one-half gallon plastic container contents are mixed by agitation. Approximately 100 mL of the raw water samples is then placed in the filter funnel. The two-way valve is turned to introduce vacuum to the separatory funnel. As soon as the sample has filtered through to the separatory funnel, the two-way valve is turned to atmosphere and the contents are used to rinse the TDP/NO_3 sample storage bottle and the Cl/Si sample

storage bottle. The empty separatory funnel stopcock is closed, the half gallon container contents mixed again by agitation and approximately 250 mL of sample are added to the filter funnel. The two-way valve is turned to put vacuum on the separatory funnel. When the sample has filtered into the separatory funnel, the two-way valve is turned to atmosphere, the separatory funnel contents are drained into the TDP/NO_3 sample storage bottle and the Cl/Si sample storage bottle. With the filter forceps, the used filter is replaced with a new filter. If there are no more samples, the cover is placed back on the filter funnel. During the filtering operation, the half gallon plastic container is mixed by agitation and the TP sample storage bottle rinsed with and then filled with raw water from the container. When all of the samples have been filtered, the bottle-top dispenser is used to add 0.4 mL of sulfuric acid to each of the TDP/NO_3 samples and TP samples in their 125 mL sample storage bottles. The technician then places the nutrient sample storage bottles in a refrigerator and records their employee ID and finish time in the Batch Log. The samples are sorted into analytical batches and placed into Ziploc bags by the GLAS contract chemist prior to being transported back to the Analytical Laboratory.

7.4 For metals samples, mix the one-half gallon raw water sample by agitation and rinse the 250mL metals storage container. Then, fill the rinsed bottle directly from the half gallon container. When all 250mL bottles are filled add 1.25mL of 1:1Nitric acid to each bottle from the bottle-top dispenser under the fume hood. The technician then stores the sample bottles in a refrigerator, records the sample ID and checks off the sample on the nutrients preparation sheet. If 250mL bottles are not available and another size bottle is used the ratio of Nitric acid to sample is 5mL to 1L.

8.0 SAFETY AND WASTE HANDLING

8.1 Refer to GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended) and individual instrument procedural operations manuals for specific details on applicable 1) personal health and safety issues; 2) instrumental, chemical, and waste handling procedures; and 3) accident prevention. This applies to all EPA personnel, EPA contractors or federal, state, or local government agencies, and persons who operate or are passengers onboard US EPA GLNPO vessels during all activities and surveys.

8.2 All containers storing reagents, standards, controls, blanks, and wastes used in the laboratory must be properly identified through appropriate labeling and hazard definition.

8.3 Every chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable. Please refer to Appendix L in GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended) for more detailed descriptions of the potential risks associated with any chemicals used in this method. It is good laboratory practice to wear a lab coat, safety goggles and gloves at all times.

8.4 It is the responsibility of the user of this method to comply with relevant chemical disposal and waste regulations as sited in GLNPO's *Health, Safety and Environmental Compliance Manual* (May 1997, or as amended). All applicable safety and waste handling rules are to be followed. Good technique includes minimizing contaminated waste.

8.5 Over-board discharges of chemical wastes are forbidden.